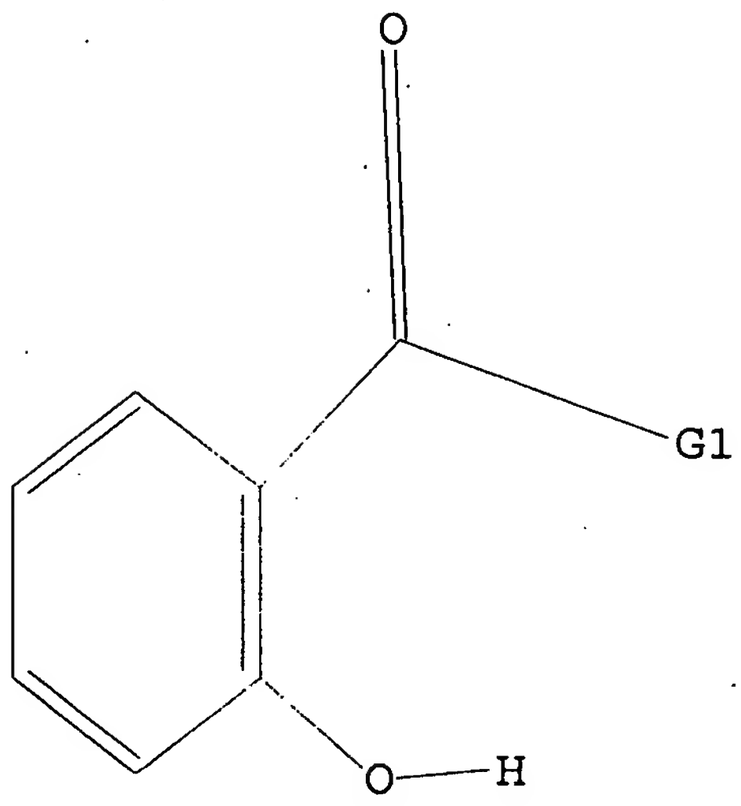
L18 ANSWER 1 OF 1 CAPLUS COPYRIGHT 2007 ACS on STN
TI Skin whitening compositions based on hydroxyaryl alkyl ketones and their
isosteric derivatives
AB Certain hydroxyaryl or polyhydroxyaryl compds. that contain an alkyl
carbon side chain with a hetero-atom group attached by a double bond at
the first carbon atom of the alkyl side chain that is directly attached to
the aromatic ring provide a surprising and unexpected skin whitening effect.
The addition of metal ions, such as copper, zinc, selenium, or vanadium and
certain antioxidant compns. addnl. increases the skin whitening effect.
For example, a skin lightener serum with copper ions was prepared by mixing
water 20.0, quinacetophenone 5.0, methylpropanediol 69.0, dimethicone
copolyol 4.0, preservatives 0.5, copper gluconate 0.5, and ammonium
acryloyidimethyltaurate/VP copolymer 1.0 weight%, resp., at room temperature The
product had a clear to slightly hazy syrup-like light blue appearance,
typical of a skin serum product. It was absorbed rapidly with a silky
smooth skin feel. A skin lightening effect was observed in human volunteers.

ACCESSION NUMBER: 2005:1293554 CAPLUS
DOCUMENT NUMBER: 144:40393
TITLE: Skin whitening compositions based on hydroxyaryl alkyl ketones and their isosteric derivatives
INVENTOR(S): Gupta, Shyam K.
PATENT ASSIGNEE(S): USA
SOURCE: U.S. Pat. Appl. Publ., 15 pp.
CODEN: USXXCO
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 2

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2005271608	A1	20051208	US 2004-862037	20040605
US 2007099886	A1	20070503	US 2006-309437	20060804
PRIORITY APPLN. INFO.:			US 2004-862037 A2	20040605

=> d que l18
L13 STR



G1 Me,Et,n-Pr,i-Pr,n-Bu,i-Bu,s-Bu,t-Bu

Structure attributes must be viewed using STN Express query preparation.
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L16 11834 SEA L15
L17 1093 SEA L16 (S) (HYDROXYARYL OR ACETOPHENONE)
L18 1 SEA L17 (S) (TOPICAL? OR LOTION? OR SKIN? OR DERMA? OR
EPIDERM? OR COSMETIC? OR COSMECEUTICAL?)

=> d his

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FILE 'REGISTRY' ENTERED AT 17:04:01 ON 08 AUG 2007
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L6 STRUCTURE UPLOADED
L7 36 S L6 SSS SAM

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L13             STRUCTURE UPLOADED
L14          43 S L13 SSS SAM
L15        8633 S L13 SSS FULL

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2007
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L17        1093 S L16 (S) (HYDROXYARYL OR ACETOPHENONE)
L18           1 S L17 (S) (TOPICAL? OR LOTION? OR SKIN? OR DERMA? OR
EPIDERM? O
L19           1 S L17 (P) (TOPICAL? OR LOTION? OR SKIN? OR DERMA? OR
EPIDERM? O
L20        1094 S L16 (P) (HYDROXYARYL OR ACETOPHENONE)
L21           1 S L20 (P) (TOPICAL? OR LOTION? OR SKIN? OR DERMA? OR
EPIDERM? O
L22          10 S L20 AND (TOPICAL? OR LOTION? OR SKIN? OR DERMA? OR
EPIDERM? O
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L24           9 S L23 NOT L18

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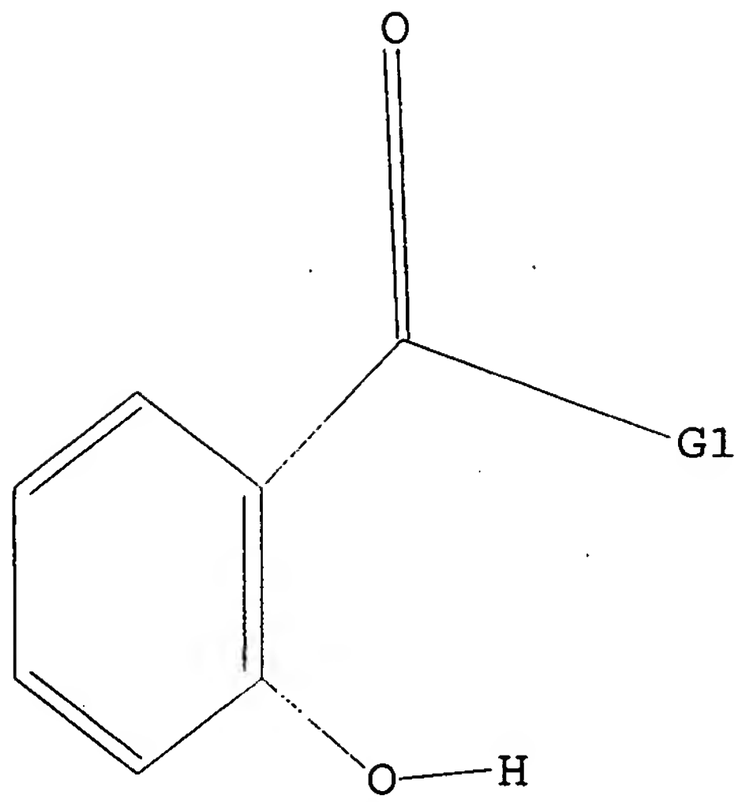

G1 Me,Et,n-Pr,i-Pr,n-Bu,i-Bu,s-Bu,t-Bu

Structure attributes must be viewed using STN Express query preparation.

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L15          8633 SEA FILE=REGISTRY SSS FUL L13
L16         11834 SEA L15
L17          1093 SEA L16 (S) (HYDROXYARYL OR ACETOPHENONE)
L19             1 SEA L17 (P) (TOPICAL? OR LOTION? OR SKIN? OR DERMA?
OR
                  EPIDERM? OR COSMETIC? OR COSMECEUTICAL?)


=> d que l21
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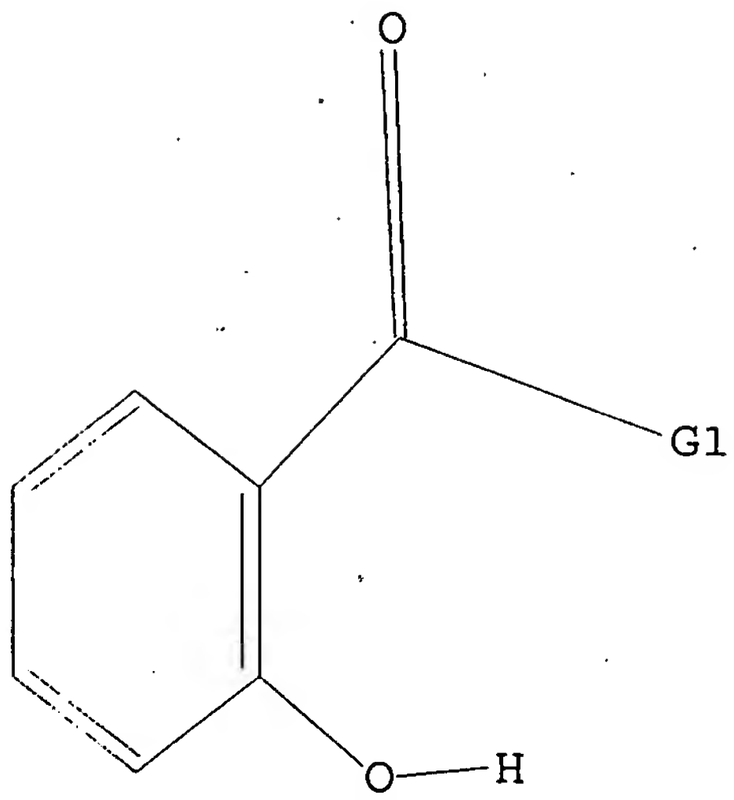

G1 Me,Et,n-Pr,i-Pr,n-Bu,i-Bu,s-Bu,t-Bu

Structure attributes must be viewed using STN Express query preparation.

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L15          8633 SEA FILE=REGISTRY SSS FUL L13
L16         11834 SEA L15
L20          1094 SEA L16 (P) (HYDROXYARYL OR ACETOPHENONE)
L21             1 SEA L20 (P) (TOPICAL? OR LOTION? OR SKIN? OR DERMA?
OR
                  EPIDERM? OR COSMETIC? OR COSMECEUTICAL?)

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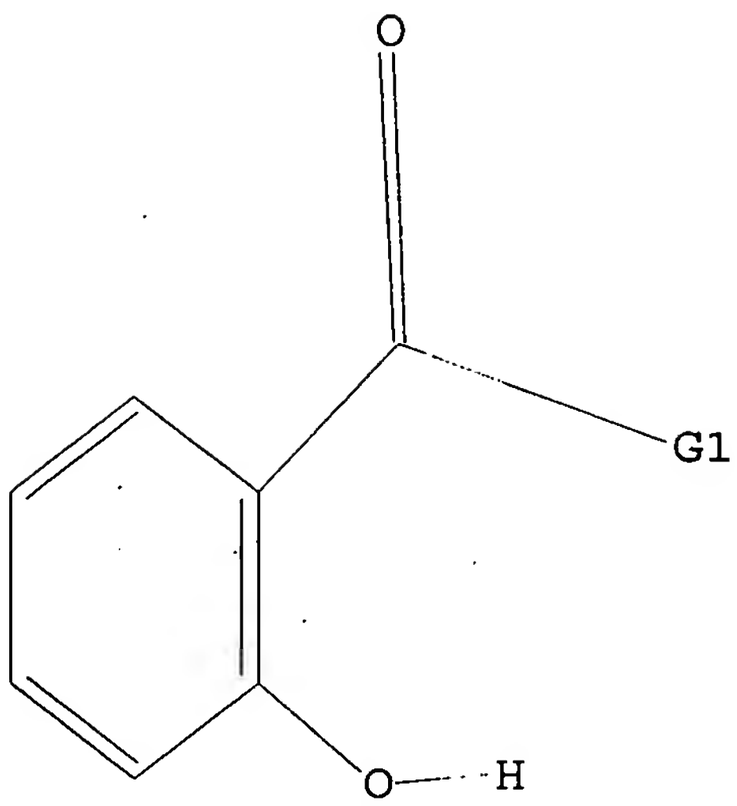


G1 Me,Et,n-Pr,i-Pr,n-Bu,i-Bu,s-Bu,t-Bu

Structure attributes must be viewed using STN Express query preparation.

L15 8633 SEA FILE=REGISTRY SSS FUL L13
L16 11834 SEA L15
L20 1094 SEA L16 (P) (HYDROXYARYL OR ACETOPHENONE)
L22 10 SEA L20 AND (TOPICAL? OR LOTION? OR SKIN? OR DERMA?
OR
EPIDERM? OR COSMETIC? OR COSMECEUTICAL?)

L24 ANSWER 1 OF 9 CAPLUS COPYRIGHT 2007 ACS on STN
TI Preparation of substituted acetophenone derivatives such as chalcones, chromones and Knoevenagel products as antitumor, antibacterial, antiviral, immunomodulating and immunostimulating agents
AB The title compds. selected from (1) chalcones R1CH:CHCOR2 [R1 = 5-hydroxymethylfuran-2-yl, 4-methoxyphenyl, 4-hydroxy-3-methoxyphenyl, furan-2-yl, 3-methoxy-4-(3-methylbut-2-enyloxy)phenyl; R2 = 4-chlorophenyl, 2-hydroxyphenyl, 2-hydroxy-5-(3-methylbut-2-enyl)phenyl, etc.], (2) chromones [I; R1 as above; R3, R4, R5 = H, 5-hydroxymethylfuran-2-yl, 4-methoxyphenyl, etc.] and (3) Knoevenagel products (CN)2C:CR6Me [R6 = 2-(3-methylbut-2-enyloxy)phenyl, 4-(3-methylbut-2-enyloxy)phenyl, 4-hydroxyphenyl], were prepared Thus, 1-(2-hydroxyphenyl)-3-[3-methoxy-4-(3-methylbut-2-enyloxy)phenyl]propenone and AcONa·3H2O in EtOH were heated under reflux for 8 h to give 83% 2-[3-methoxy-4-(3-methylbut-2-enyloxy)phenyl]chroman-4-one. The latter showed very strong activity against a variety of human tumor cells with IC50 = 6-84 μg/mL.

ACCESSION NUMBER: 2007:322971 CAPLUS
DOCUMENT NUMBER: 146:358706
TITLE: Preparation of substituted acetophenone derivatives such as chalcones, chromones and Knoevenagel products as antitumor, antibacterial, antiviral, immunomodulating and immunostimulating agents
INVENTOR(S): Suarez, Jose Augustin Quincoces; Peseke, Klaus; Molina Ruiz, Reinaldo
PATENT ASSIGNEE(S): Riemser Arzneimittel AG, Germany
SOURCE: Eur. Pat. Appl., 29pp.
CODEN: EPXXDW
DOCUMENT TYPE: Patent
LANGUAGE: German

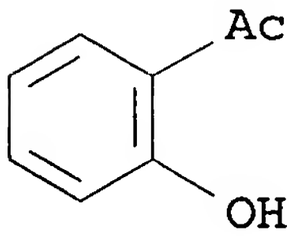
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 1764363	A2	20070321	EP 2006-19204	20060913
EP 1764363	A3	20070516		

R: AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LI, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR, AL, BA, HR, MK, YU

DE 102005044156	A1	20070329	DE 2005-102005044156	20050915

PRIORITY APPLN. INFO.: DE 2005-102005044156A 20050915
OTHER SOURCE(S): CASREACT 146:358706; MARPAT 146:358706
IT **118-93-4**
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of substituted **acetophenone** derivs. such as chalcones, chromones and Knoevenagel products as antitumor, antibacterial, antiviral, immunomodulating and immunostimulating agents)
RN 118-93-4 CAPLUS
CN Ethanone, 1-(2-hydroxyphenyl)- (CA INDEX NAME)



L24 ANSWER 3 OF 9 CAPLUS COPYRIGHT 2007 ACS on STN
TI Dihydro and tetrahydrofurans
AB Compds. RR1C.O.CR2R3.Z, in which Z is an ethylene or vinylene radical which may be substituted by a carboxyl, alkyl or ether group, R is the

group COCYHCOX in which Y is H or lower hydrocarbon, X is H or lower
hydrocarbon such as cycloalkyl, monocyclicaryl, dialkylaminoalkyl or a
heterocyclic group, R1 and R2 are lower alkoxy and R3 is H or lower alkyl,
are useful as intermediates in the preparation of polyhydroxyphenyl ketones.
The latter are useful as tanning agents, in permanent inks, as photog.
developers, as chelating agents and as sunscreen agents. Et furoate (I)
38.0 parts and I volume concentrated H2SO4 in 250 vols. MeOH is electrolyzed in a
Ni cathode-graphite anode cell, 19.4 amp. hrs. being passed in 8 h.
beginning at 9 v. and 3.5 amp. at -15° to -22°. The solution
is neutralized with NaOMe in MeOH, concentrated, filtered and the filtrate
distilled to give Et 2,5-dimethoxy-2,5-dihydrofuroate (II), b21
136-9°, nD25 1.4480. II 25.9 and NaH 6.8 are treated with acetone
(III) 16.5 in 25 vols. Et2O added dropwise over 0.5 h. at 30-40°.
H is evolved and stirring continued 0.5 h., 100 vols. Et2O added and the
mixture kept 3 days. EtOH (95%, 15 vols.) is added, and the mixture decomposed
at 0° with 100 vols. water and AcOH 18 parts. The layers are
separated, the aqueous phase extracted with Et2O, and the combined layers are dried
and distilled to give
2-acetoacetyl-2,5-dimethoxy-2,5-dihydrofuran (IV), b0.2
97°, nD23 1.4956. Me 2,5-dimethoxytetrahydrofuroate (V) 26.1 and
NaH 6.6 treated with III 16 parts give 2-acetoacetyl-2,5-
dimethoxytetrahydrofuran (VI), b0.5 87-91°, nD23 1.4825. IV 5.35
parts, 50 vols. 0.1N HCl and a small chip of solid CO2 are stirred 6 h. in
a closed flask.
2,2',4,4',5,5'-Hexahydroxy-3,3'-diacetyldiphenyl, m.
280° (decomposition), seps. as a mustard-yellow precipitate and is filtered
off. Chilling the clear yellow filtrate gives brilliant yellow crystals

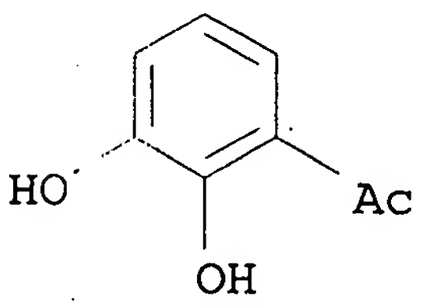
of 2,3,6-trihydroxyacetophenone (VII), m. 157.5-59°. VI 7.4 parts
and 75 vols. 0.1N HCl refluxed 1 h. give 2,3-dihydroxyacetophenone, m.
98-8.5°, yellow. V 38.0, NaH 4.8, and 3-acetylpyridine 12.1 parts
give 2-nicotinoylacetyl-2,5-dimethoxytetrahydrofuran (VIII), b0.06
150-8° (slight decomposition), viscous yellow oil which solidifies after
several months. VIII 8.5 parts and 200 vols. 0.5N HCl give
3-nicotinoylpyrocatechol, m. 154-6°, yellow. I 38.0 is
electrolyzed in 250 vols. EtOH to give Et 2,5-diethoxy-2,5-dihydrofuroate
(IX) 31.6 parts, b21 145-50°. IX condensed with III 16.5 and Nail
6.8 parts gives 2-acetoacetyl-2,5-diethoxy-2,5-dihydrofuran (X).
Hydrolysis of X with HCl gives VII. V 38.0, NaH 4.8, and
diethylaminoacetone 25.8 parts give 2-ethyldiaminoacetoacetyl
2,5-dimethoxytetrahydrofuroate. V 38.0, NaH 4.8, and Me cyclohexyl ketone
24.6 parts give 2-(cyclohexanecarbonylacetyl)-2,5-
dimethoxytetrahydrofuran. Me 2,5-dimethoxy-5-isopropyltetrahydrofuroate
31.6, NaH 6.8, and III 16.5 parts give 2-acetoacetyl-5-isopropyl-2,5-
dimethoxytetrahydrofuran, b0.1 93-7°. V 25.9, NaH 6.8, and
pinacolone 28.5 parts give 2-pivaloylacetyl-2,5-dimethoxytetrahydrofuran,
b0.1 105-12°. V 25.9, NaH 6.8, and 4-methylpropiophenone 45.0
parts give 2-[α-(4-methylbenzoyl)propionyl]-2,5-
dimethoxytetrahydrofuran. V 38.0 NaH 4.8, and PhCOMe 24 parts give
2-benzoylacetyl-2,5-dimethoxytetrahydrofuran which can be hydrolyzed to
2,3-dihydroxybenzophenone, m. 65°.

ACCESSION NUMBER: 1960:39125 CAPLUS
DOCUMENT NUMBER: 54:39125
ORIGINAL REFERENCE NO.: 54:7732f-i,7733a-d
TITLE: Dihydro and tetrahydrofurans
INVENTOR(S): Boehme, Werner R.
PATENT ASSIGNEE(S): Ethicon, Inc.
DOCUMENT TYPE: Patent

LANGUAGE: Unavailable
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2921078		19600112	US 1958-757451	19580827

IT **13494-10-5P**, **Acetophenone**, 2',3'-dihydroxy-
RL: PREP (Preparation)
(preparation of)
RN 13494-10-5 CAPLUS
CN Ethanone, 1-(2,3-dihydroxyphenyl)- (CA INDEX NAME)



L24 ANSWER 5 OF 9 CAPLUS COPYRIGHT 2007 ACS on STN
TI Rearrangements of phenol esters in hydrogen fluoride. Condensation of
β-oxo esters with phenols in HF
AB Results of Fries rearrangements of phenol esters induced by anhydrous HF are
compared with those reported earlier using BF3 and AlCl3. The esters are
mixed with com. anhydrous HF at 0° in a cast-steel bomb of 360 mL.
capacity, then allowed to warm to room temperature or heated to the reaction
temperature and rocked; after the desired reaction period, the HF is driven off
at 100° and the residue poured into a little H2O; if not crystalline,
the product is taken up in Et2O. Of a variety of conditions, the best (10

g. PhOAc and 150 mL. HF kept 24 h. at 20°) gave an 81% yield of p-hydroxyacetophenone (I), m. 105-7° (2,4-dinitrophenylhydrazone, dark red); small amts. (2%) of the o-isomer (II) were isolated by steam distillation and converted to the light red 2,4-dinitrophenylhydrazone. Heating 10 g. PhOH, 10 g. HOAc, and 150 mL. HF 1 h. at 100° gave 61.6% I (m. 107-9°) and 2% crude II; 24 h. at 20° gave 40% I. Reaction of 10 g. m-MeC6H4OAc in 20 mL. HF for 24 h. at 20° gives 16.5% 4-hydroxy-2-methylacetophenone (III), non-volatile in steam, plates, m. 125°, and 74% steam-volatile 7-hydroxy-4-methylacetophenone (IV), b15 115°, m. 20-1°; 1 h. at 100° gives 8.5% III and 79.5% IV. Heating 17.8 g. 2,3,5-trimethylphenyl acetate with 100 mL. HF 1 h. at 100° yields 14.6 g. crude product from which 84% 2,3,5-trimethylphenol is isolated by crystallization from H2O, m. 95-6° (from petr. ether); the crude product yields traces of a red, unidentified 2,4-dinitrophenylhydrazone. The product obtained by heating 15 g. p-MeC6H4OAc in 30 mL. HF for 1 h. at 120-5° is distilled (b16 124°) and recrystd. from petr. ether to give 63% 2-hydroxy-5-methylacetophenone. Reaction of 2-MeOC6H4OAc under the best (100 g. in 200 mL. HF for 40 days at 0°) of a variety of conditions gives 78% of a mixture of 4-hydroxy-3-methoxyacetophenone (V) and 3-hydroxy-4-methoxyacetophenone (VI), b1.5 152-9°, m. 72-96°, shown by reference to a mixed-m.p. diagram of authentic V (m. 116°) and VI (m. 93°) to consist of 64% V and 36% VI; the diagram shows a eutectic at 40% V, which m. 72°. After heating 25 g. V in 20 mL. HF for 45 min. at 70°, only 0.7 g. of unchanged V was isolable. Reaction of 12.5 g. guaiacol with 8 g. glacial HOAc in 200 mL. HF for 40 days at 0° gives 63% of a mixture, m. 72-93° (60% V and 40% VI), shown by paper chromatog. (cf. Way and Gailey, C.A. 45, 10498b) to be free of 1-hydroxy-2-methoxyacetophenone. After 24 h. at

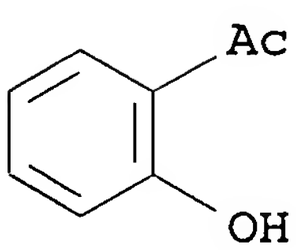
room temperature in 150 mL. HF, 5 g. Ph chloroacetate yields a product from
which 0.5 g. red crystals are isolated by crystallization from MeOH; these give a
precipitate on treatment with a solution of 2,4-dinitrophenylhydrazine; the alc.
mother liquors produce a strongly-itching allergic exzema on contact with
the **skin** and were not investigated further. After standing 20
days in HF, Ph trichloroacetate is recovered essentially unchanged;
complete decomposition results on heating 1 h. at 100°. Guaiacyl
formate (nD20 1.523, prepared in 80% yield by the action of 15% HO2Ac in
glacial HOAc on o-MeOC6H4CHO) reacts with HF to yield guaiacol and CO as
principal products; no appreciable carbonylic material is formed.
(PhO2C)2 fails to react in HF at room temperature and is decomposed at higher
temps., presumably due in part to its low solubility in HF; heating 5 g. of the
ester with 20 mL. CCl4 and 40 mL. HF at 80° for 4 h. results in
formation of 0.3 g. 4,4'-dihydroxybenzophenone (presumably attack on the
CCl4), yellow needles, m. 206°. From the product formed by shaking
10 g. di-Ph adipate in 100 mL. HF for 2 days is isolated 2 g.
citron-yellow microneedles (from EtOH), m. 241-2° (decomposition);
2,4-dinitrophenylhydrazone (from dioxane), m. 265-6°, analyses of
which, however, are not in agreement with those expected from the
anticipated 1,6-bis(p-hydroxyphenyl)-1,6-hexadione; the substance gives no
FeCl3 test, but turns blue-green on treatment with NH3H7P(MO2O7)6.
Recrystn. from C6H5CH3 of the yellow-red crude product from reaction of 20
g. Ph cinnamate in 40 mL. HF for 43 h. at room temperature gives a 55% yield of
4'-hydroxychalcone, yellow needles, m. 172-3°, color tests with
FeCl3 and H7P(MO2O7)6 neg. With the p-position of the Ph cinnamate
blocked, ring-closure (rather than migration of the cinnamyl group)

predominates; thus the crude product obtained by heating 10 g. p-tolyl
cinnamate (VII) in 20 mL. HF for 2 h. at 100° is distilled (b15 180-220°) and recrystd. from MeOH to give 3.2 g. 6-methyl-4-phenyl-2,3-dihydrocoumarin (VIII), prisms, m. 80-1°.
Heating 5 g. 2'-hydroxy-5'-methylchalcone (the alternate product which
might have been expected from the action of HF on VII) with 15 mL. HF for
1 h. at 100° gives a crude product which is distilled and recrystd. to
yield 1.5 g. 6-methylflavanone, plates, m. 105°. Heating 585 mg. VIII with 290 mg. freshly-prepared Pd-black for 6.5 h. at 240°, followed by distillation, gives 200 mg. colorless oil, b3 110-40° (probably 1-(m-methylphenyl)-1-phenylethane), and a viscous yellow oil (b3
160-80°) which solidifies, colorless prisms from MeOH, m. 131-2°, identified by mixed m.p. as 6-methyl-4-phenylcoumarin.
Hydrolysis of 1 g. VII (by refluxing 5 h. with 1.2 g. NaOH in 9 mL. MeOH)
gives 0.75 g. 3-(2-hydroxy-5-methylphenyl)-3-phenylpropionic acid, m.
125° (from C6H6), which loses H2O at 140° and is thus reconverted to VIII. Treating VIII (250 mg.) with aqueous KOH and Me2SO4
yields a mixture of acidic and neutral products; recrystn. of the former
from MeOH gives 0.75 g. 3-(2-methoxy-5-methylphenyl)-3-phenylpropionic
acid, needles, m. 136-7°; the neutral material is presumed to be the corresponding Me ester, prisms from aqueous MeOH, m. 51-2°. Using
the procedure of Spasov (C.A. 36, 7010.2), 15 g. phenylpropiolyl chloride
gives 18 g. p-tolyl phenylpropiolate (IX), needles from petr. ether, m.
56-7°. IX (10 g.) in 150 mL. HF for 5.5 h. at 0° gives a crude product which is distilled to give 3.2 g. viscous yellow oil, b1.5
140-80°, which solidifies; recrystn. from MeOH gives 2.2 g. yellow
prisms (IXa), m. 130-1°; mother liquors yield an addnl. 0.5 g. IXa.

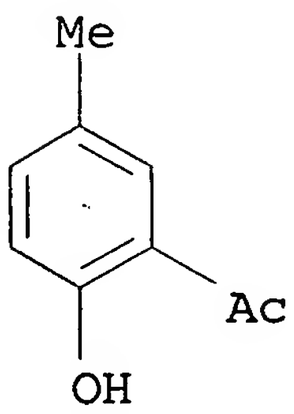
By standing overnight in 5% MeOH-KOH, the yellow impurity is destroyed,
leaving colorless 6-methyl-4-phenylcoumarin, m. 130-1°.
Condensation of β-oxo esters with phenols in the presence of HF gives
good yields of coumarins or flavones. The crude product formed by heating
10 g. EtO2CCH2Ac (X) with 10 g. PhOH in 200 mL. HF for 1 h. at 100°
is taken up in Et2O, washed with 2M NaOH and H2O, distilled, and recrystd.
from H2O, giving 7.5 g. 4-methylcoumarin, m. 81-2°. p-Cresol (XI)
reacts similarly, giving 65% 4,6-dimethylcoumarin, prisms from MeOH, m.
149-51°. After heating 5 g. EtO2CCH2Bz (XII) with 6 g. XI in 110 mL. HF for 1 h. at 100°, the product is distilled and the viscous main
fraction (b0.8 160-95°) dissolved in 5% MeOH-KOH and let stand 12 h. Strong dilution with H2O gives a precipitate which is recrystd. from aqueous MeOH
and from petr. ether to yield 1.5 g. 6-methylflavone, needles, m. 122°; following acidification of the filtrate from the 1st, a 2nd precipitate slowly forms which is recrystd. from aqueous MeOH to give 1 g.
6-methyl-4-phenylcoumarin, prisms, m. 131°. Similarly, PhOH and XII react to give flavone, m. 96-7°, and 4-phenylcoumarin, m. 91-2°.

ACCESSION NUMBER: 1956:12308 CAPLUS
DOCUMENT NUMBER: 50:12308
ORIGINAL REFERENCE NO.: 50:2566c-i,2567a-h
TITLE: Rearrangements of phenol esters in hydrogen fluoride.
Condensation of β-oxo esters with phenols in HF
AUTHOR(S): Dann, Otto; Mylius, Gering
CORPORATE SOURCE: Univ. Erlangen, Germany
SOURCE: Ann. (1954), 587, 1-15
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 50:12308
IT **118-93-4P**, **Acetophenone**, 2'-hydroxy- **1450-72-2P**, **Acetophenone**, 2'-hydroxy-5'-methyl- **6921-64-8P**, **Acetophenone**, 2'-hydroxy-4'-methyl-

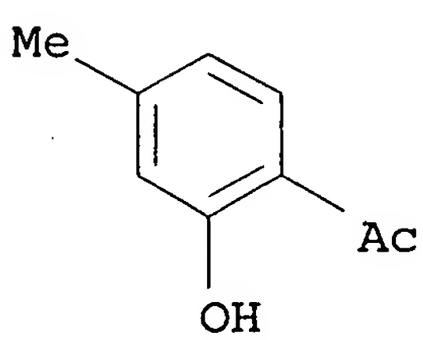
RL: PREP (Preparation)
(preparation of)
RN 118-93-4 CAPLUS
CN Ethanone, 1-(2-hydroxyphenyl)- (CA INDEX NAME)



RN 1450-72-2 CAPLUS
CN Ethanone, 1-(2-hydroxy-5-methylphenyl)- (CA INDEX NAME)



RN 6921-64-8 CAPLUS
CN Ethanone, 1-(2-hydroxy-4-methylphenyl)- (CA INDEX NAME)



L24 ANSWER 8 OF 9 CAPLUS COPYRIGHT 2007 ACS on STN
TI Synthesis of some hydroxy alkylbenzoquinones
AB The Thiele addition of 30 mL. Ac2O containing a little 96% H2SO4 to 4.5 g.
cyclohexyl-p-quinone (I) was incomplete after 27 days; the excess Ac2O was

hydrolyzed in a large volume of H2O and the product extracted with ether; the ether residue gave 4.55 g. 2-hydroxy-5-cyclohexyl hydroquinone triacetate (II), m. 117.8-18.2° (from C6H6ligroine). II (1.67 g.) in 15 mL. EtOH (flushed with pure N) treated with 1.5 g. NaOH in 10 mL. H2O (flushed with N), the mixture warmed 0.5 h. on a steam bath, acidified with 8:1 HCl, cooled, and treated with 5.4 g. FeCl3.6H2O in 20 mL. dilute HCl gave 0.86 g. 2-hydroxy-5-cyclohexyl-p-quinone (III), m. 89.5-91.5° (by sublimation at 65-75°/0.1 mm.), sensitive to all solvents; alkaline solns. are red and do not change on addition of NCCH2CO2Et (Craven test); the solution in concentrated H2SO4 is orange and stains the **skin** deep blue.

Resorcinol (22.0 g.) and 22.0 g. cyclohexanol mixed with 2.0 g. Superfiltrol X-365 D were heated till a mixture of H2O and cyclohexane began to distill, the H2O separated, the organic layer returned till 3.5 mL. H2O was collected and the temperature rose to 175°, C6H6 added to the cooled mixture, the catalyst filtered, and the residue distilled to give 35.5 g. light yellow oil yielding from C6H6-ligroine 32.5 g. 4-cyclohexylresorcinol (IV), m. 123.5-5.5°. BF3 (1.5 g.) acetylation (60 g. glacial HOAc) of 3.85 g. IV overnight and 3 h. on a steam bath gave 3.67 g. 4-cyclohexyl-6-acetylresorcinol (V), m. 144.5- 5.2° (from C6H6-ligroine). To 2.34 g. V in 20 mL. N NaOH, flushed with pure N, was added 21.2 mL. 0.71 M H2O2, the mixture kept 1 h. at room temperature, then warmed briefly, cooled, acidified (some insol. material filtered off), extracted twice with ether (which gave a little dark oil), and acetylated in the presence of a little Zn to give a small amount of II, m. 116-17.5°.

To a stirred and cooled solution (-5° to 5°) of 9.61 g. IV in 175 mL. 95% EtOH containing 58.5 g. HCl was added 3.6 g. $NaNO_2$ in 20 mL. H_2O; dilution after 1.5 h. with 1 l. H_2O gave 8.98 g. 6-nitroso-4-cyclohexylresorcinol (VI), decompose 195° (from Me_2CO). Small portions of solid $NaHSO_3$ were added to 1.1 g. VI in 50 mL. 0.5 N NaOH till the red color was discharged; a white solid neutralization with HOAc precipitated aminoresorcinol (VII), which was filtered, washed with fresh dilute $NaHSO_3$, and dissolved in 40 mL. dilute HCl containing a little $SnCl_2$; filtration over a pad of Darco gave a colorless solution but no crystalline HCl salt; the solution was used as such for oxidation expts. Hydrogenation of 1.1 g. VI in HOAc and Ac_2O with 70 mL. presatd. PtO_2 was complete in 20 min., giving 0.8 g. 6-acetamido-4-cyclohexylresorcinol (VIII), m. 204.2-5.2° (from $CHCl_3$). From 30 mL. of a warm solution containing 0.003 mol VII.HCl treated with $FeCl_3$ was obtained a green oil, extraction of which gave only 0.03 g. III, m. 84-7° (sublimed at 0.2 mm.). VIII in HOAc with excess $FeCl_3$, on ether extraction and sublimation at 90°/0.2 mm. gave a trace of III, m. 88.5-90°. No III was obtained from VIII with HNO_3 or chromic acid.

VI (as the quinone monooxime) was hydrolyzed directly by heating 0.73 g. to reflux a few min. in 10 mL. dioxane with 0.5 mL. Me_2CO 0.5 g. CuO, and 1.6 mL. HCl diluted with 2.0 mL. H_2O; dilution gave a dark red solid, which, dried and sublimed at 80°/0.2 mm., yielded 0.3 g. III, m. 87.5-9.5°. (4-Cyclohexylbutyl)p-quinone (IX) (1.23 g.) in 20 mL. Ac_2O containing a little H_2SO_4 gave a neg. quinone test after 24 h. at room temperature; the mixture stirred with H_2O yielded 1.2 g. 2-hydroxy-5-(4-cyclohexylbutyl)hydroquinone triacetate (X), m. 77.0-7.7° (from ligroine or dilute EtOH). The filtrate from X on concentration gave a 2nd solid,

m. 62-3°, not identified, which did not have the composition of an isomeric triacetate. X (1.95 g.) in EtOH was hydrolyzed by heating 0.5 h. (in a N atmospheric) with dilute NaOH, acidified, cooled, treated with sufficient EtOH to dissolve the oil, then with excess alc. FeCl3, and the oily product separated, dried, and sublimed at 60°/1 mm. to give 0.83 g.

2-hydroxy-5-(4-cyclohexylbutyl)-p-quinone (XI), microcryst. solid, m. 103.5-5.5°, dissolves in dilute NaOH with red color, gives no Craven test (C.A. 25, 4538), forms in concentrated H2SO4 a yellow solution which stains the **skin** deep blue. Decomposition with NaOAc of the red complex formed by 22 g. resorcinol, 42.5 g. cyclohexanebutyric acid, and 20.2 g. BF3 during 12 h. at room temperature and 3 h. on a steam bath gives 43.4 g.

4-(4-cyclohexylbutyryl)resorcinol (XII), m. 98-107° (2 crystns. from C6H6-ligroine); the sharpness of the m.p. could not be increased by further recrystn. or fractionation on a column of acid-washed alumina.

The diacetate, from Ac2O and NaOAc, m. 82.2-3.2°, gave on hydrolysis XII with the same unsharp m.p. Reduction of XII by the Clemmensen-Martin procedure was complete in 8 h., giving an oil, b0.2 160-70°, rapidly solidifying to white 4-(4-cyclohexylbutyl)resorcinol (XIII), m. 83.5-4.5°; diacetate, m. 58-8.6° (from dilute EtOH). BF3 acetylation of 5.0 g. XIII as for the preparation of V gave 4.73 g. yellow-white 4-(4-cyclohexylbutyl)-6-acetylresorcinol (XIV), m. 71.5-2.2° (from C6H6-ligroine); diacetate, white needles, m. 45.7-6.7°. The modified Dakin oxidation of 2.9 g. XIV as for V gave a dark uncrystallizable oil. Acetylation with a little Zn dust present gave a few white needles, m. 76.0-7.2°,

identical (mixed m.p.) with X, thus establishing the structure of X.

Addition of 35 g. Ac2O containing 2% H2O to 3.9 g. 2-methyl-5-(4-cyclohexylbutyl)-

p-quinone (XV) was completed in 1 h. and after 12 h. the mixture gave 2.96

g. 2-hydroxy-3-methyl-6-(4-cyclohexylbutyl)hydroquinone triacetate (XVI),

m. 83.0-3.5° (from ligroine). Filtrates from several expts. gave a

dark yellow viscous oil which did not crystallize even after evaporative

distillation at 170°/0.3 mm. Alkaline hydrolysis of 1.25 g. XVI under N,

dilution, and acidification gave 0.8 g. 2-hydroxy-3-methyl-6-(4-cyclohexylbutyl)hydroquinone (XVII), m. 106-7° (brown melt) (from C6H6 ligroine). Oxidation of 0.49 g. XVII in 15 mL. 95% EtOH with excess

alc. FeCl3 gave 0.43 g. of the p-quinone (XVIII), sublimes at 80°/0.1 mm., m. 109.5-111.5°, gives a reddish brown color with 96% H2SO4, does not stain the **skin**. To establish the structure of XVIII, 8 mL. of 0.28 M Ac2O2 solution in HOAc and 0.56 g. XVIII

in HOAc were heated 1 h. on a steam bath, cooled, diluted, and the yellow

oil extracted with ether; the ether residue in petr. ether precipitated an oily

crystalline solid (probably starting material), which was filtered off and the

filtrate evaporated and reductively acetylated with Ac2O, Zn dust, and a

little NaAOc, giving a small amount of light yellow oil which, crystallized from

ligroine-petr. ether, yielded 2-hydroxy-3,5-dimethyl-6-(4-cyclohexylbutyl)hydroquinone triacetate (XIX), m. 109.0-10.6°; hydrolysis, oxidation, and 2 recrystns. from dilute HOAc gave 2-hydroxy-3,5-dimethyl-6-(4-cyclohexylbutyl)-p-quinone (XX), m. 63.5-4.5°. 2,6-Dimethyl-3-(4-cyclohexylbutyl)-p-quinone (0.8 g.) reacted slowly with 10 mL. Ac2O containing a few drops H2SO4; in 44 h. the

quinone test was neg. and the mixture stirred with H2O gave 1.17 g. solid

XIX, m. 110.5-11.0° (from ligroine). Alkaline hydrolysis of 4.18 g.

XIX under N and acidification gave 2.42 g. hydroquinone, m. 115-15.6°. This (2.0 g.) in 15 mL. HOAc was oxidized with excess FeCl3 in HOAc and the mixture diluted to give 1.73 g. XX, m. 65.0-5.6° [from petr. ether, (b. 20-40°) or sublimation at 55-60°/0.07 mm.]. XX is not extracted from the ether by saturated NaHCO3 or 10% Na2CO3 but is extracted by dilute NaOH as a violet Na salt, gives a deep violet color with H2SO4, and does not dye the **skin**. 2,3-Dimethyl-5-(4-cyclohexylbutyl)-p-quinone reacted slowly with Ac2O containing H2SO4; after 26 h. quinone was still present. The solution stirred with H2O and the residual oil extracted with ether gave a gum which was passed in ligroine solution through a 5-in. column of acid-washed alumina; the poorly adsorbed unreacted quinone was eluted by ligroine, and further elution with C6H6 and ligroine gave 3 fractions, all of which, crystallized from ligroine, gave 0.3 g. 2-hydroxy-3-(4-cyclohexylbutyl)-5,6-dimethylhydroquinone triacetate (XXI), m. 74.5-5.5° (from ligroine). XXI (150 mg.) in EtOH hydrolyzed under N with dilute NaOH gave on acidification an amber oil which solidified, and in warm HOAc with 1 g. FeCl3 yielded 105 mg. p-quinone, sublimes at 60°/0.2 mm., m. 68.5-70.0°, forms a sparingly soluble deep violet Na salt, gives a deep violet color in concentrated H2SO4. A suspension of 4.4 g. 2,5-di-tert-butyl-p-quinone in 40 mL. Ac2O containing H2SO4, kept 48 days with occasional warming on a steam bath, gave a semisolid residue, which, extracted with ether, yielded 3.2 g. white prisms, m. 110-11°, resolidified at 115° and remelted at 121-2°; anal. (C 62.85, 62.61; H 6.39, 6.30; calculated for C16H20O6 62.53, H 6.23%) indicated that one of the tert-Bu groups had been eliminated. A suspension of the diazonium salt from 52.5 g. p-H2NC6H4SO3H.2H2O was added to 200 g. ice and 30.5 g. 2,5-xylenol and 55

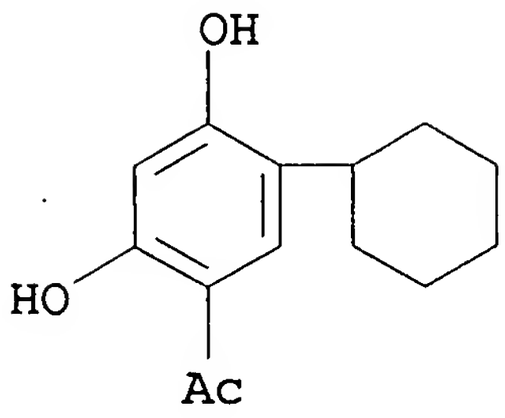
g. NaOH in 300 mL. H2O, the deep red solution warmed to 40-50° after
1.5 h., treated with a slight excess of NaHSO3, the cooled suspension
filtered, the yellow aminophenol washed with fresh dilute NaHSO3,
transferred to a mixture of 32 mL. HCl, 250 mL. H2O, and 1 g. SnCl2, heated
to boiling, 50 mL. HCl added, the mixture filtered over a pad of Darco, and
the filtrate treated with 100 mL. HCl to give on cooling 32.9 g.
2,5,4-Me2(H2N)C6H2OH.HCl; this (31.8 g.) in 50 mL. H2SO4 and 1000 mL. H2O,
added all at once to 35 g. K2Cr2O7 in 500 mL. H2O gave 23.5 g. bright
yellow 2,5-dimethyl-p-quinone (XXII), m. 123-4°. XXII (13.6 g.)
after 11 h., in 100 g. Ac2O and 3 g. 96% H2SO4 at 40-50° gave a
neg. quinone test, and addition to 600 mL. H2O precipitated 26.5 g.
2-hydroxy-3,6-dimethylhydroquinone triacetate (XXIII), m. 104-4.6°
(from C6H6-ligroine). XXIII (14 g.) was hydrolyzed under N with 15 g.
NaOH in dilute EtOH; acidification and addition of more alc. gave a clear light
yellow solution to which was added 54 g. FeCl3.6H2O in acidic EtOH; dilution
gave 5.7 g. 2-hydroxy-3,6-dimethyl-p-quinone (XXIV), m. 102-4°
(decomposition) (sublimed at 80°/0.15 mm.). Cyclohexanebutyryl chloride
(22.6 g.) in 50 mL. absolute ether was shaken with ice and Na2O2 [from 17.2
mL. "Superoxol" (0.132 mol by titration) and a dilute solution of 9.6 g. NaOH],
shaking continued 10 min., and the ether layer washed with saturated salt
solution, dried over anhydrous MgSO4, filtered, and diluted with 100 mL. dry
ether; a 1-mL. aliquot titrated iodometrically indicated the presence of
0.0405 mol bis(γ-cyclohexylbutyryl) peroxide. Alkylation of freshly
sublimed 4.56 g. XXIV in 75 mL. HOAc with this peroxide solution gave a red

oil, which was washed in ether with 3 portions saturated NaHCO3 to remove unreacted XXIV as the deep violet Na salt; the ether residue, crystallized from petr. ether (b. 20-40°) at Dry-Ice temperature yielded 2.62 g. orange 2-hydroxy-3,6-dimethyl-5-(3-cyclohexylpropyl)-p-quinone, m. 80.0-0.7° (from petr. ether or dilute EtOH), gives a deep violet Na salt with NaOH. XXIV (3.04 g.) in AcOH was heated 1 h. on a steam bath with 8.89 g. com. lauroyl peroxide, the HOAc distilled in vacuo, and unalkylated quinone removed from the partially crystalline residue by extraction with NaHCO3; the residue from ligroine gave 2.68 g. 2-hydroxy-3,6-dimethyl-5-hendecyl-p-quinone, m. 102-2.3° (from ligroin, then dilute HOAc), forms a deep violet Na salt from dilute NaOH.

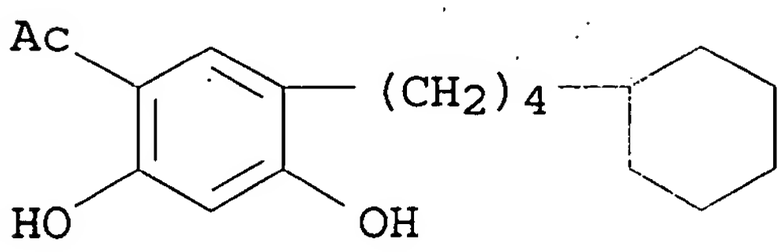
ACCESSION NUMBER: 1952:8525 CAPLUS
DOCUMENT NUMBER: 46:8525
ORIGINAL REFERENCE NO.: 46:1499g-i,1500a-i,1501a-i,1502a-b
TITLE: Synthesis of some hydroxy alkylbenzoquinones
AUTHOR(S): McLamore, W. M.
CORPORATE SOURCE: Harvard Univ.
SOURCE: Journal of the American Chemical Society (1951), 73, 2225-30
CODEN: JACSAT; ISSN: 0002-7863
DOCUMENT TYPE: Journal
LANGUAGE: Unavailable
OTHER SOURCE(S): CASREACT 46:8525

IT **159977-36-3P**, **Acetophenone**, 5'-cyclohexyl-2',4'-dihydroxy- **709032-55-3P**, **Acetophenone**, 5'-(4-cyclohexylbutyl)-2',4'-dihydroxy-
RL: PREP (Preparation)
(preparation of)

RN 159977-36-3 CAPLUS
CN Ethanone, 1-(5-cyclohexyl-2,4-dihydroxyphenyl)- (9CI) (CA INDEX NAME)



RN 709032-55-3 CAPLUS
CN Acetophenone, 5'-(4-cyclohexylbutyl)-2',4'-dihydroxy- (5CI) (CA INDEX
NAME)



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Time: 08:00:38

Inventor Name Search Result

Your Search was:

Last Name = GUPTA
First Name = SHYAM

Application#	Patent#	Status	Date Filed	Title	Inventor Name
08189799	5554999	150	02/01/1994	COLLAPSIBLE FLAT ANTENNA REFLECTOR	GUPTA, SHYAM
08502692	Not Issued	161	07/14/1995	PASSIVE ANTENNA REFLECTOR	GUPTA, SHYAM
10605191	Not Issued	61	09/14/2003	Baby Care Skin Protectant Compositions for Diaper Rash	GUPTA, SHYAM K
10708093	Not Issued	30	02/07/2004	Skin Darkening (Sunless Tanning) Compositions Based on Enhancement of Melanin Synthesis by Tyrosinase Promoters	GUPTA, SHYAM K
10908816	Not Issued	30	05/27/2005	New Ubiquitin-Proteasome Regulating Compounds and Their Application in Cosmetic and Pharmaceutical Formulations	GUPTA, SHYAM K
11161511	Not Issued	30	08/05/2005	Topical Deodorant Compositions Based on Hydroxycitric Acid	GUPTA, SHYAM K
11161575	Not Issued	30	08/08/2005	Stripless Depilatory Compositions	GUPTA, SHYAM K
11161856	Not Issued	30	08/19/2005	Sebum Control Compositions Based on Saponins and Sapogenins	GUPTA, SHYAM K
11162209	Not Issued	30	08/31/2005	Water Washable Hair Removal (Depilatory) Compositions	GUPTA, SHYAM K
11309437	Not Issued	30	08/04/2006	Multifunction "Crown Complexes" from Amino Acids and Peptides for Skin and Hair Restoration	GUPTA, SHYAM K
11670942	Not Issued	30	02/02/2007	Treatment of Topical Discomforts Including Acne, Sunburn, Diaper Rash, Wound, Wrinkles and Dandruff/Hair Loss by Natural Lignans via Fatty Acid Desaturase Inhibition	GUPTA, SHYAM K
10223671	Not	161	08/16/2002	Vitamin C stabilized topical	GUPTA, SHYAM

	Issued			formulation	K.
10248508	Not Issued	161	01/24/2003	Topical Nutraceutical Compositions with Selective Body Slimming and Tone Firming Antiaging Benefits	GUPTA, SHYAM K.
10248691	Not Issued	161	02/10/2003	Topically Bioavailable Acne and Rosacea Treatment Compositions	GUPTA, SHYAM K.
10248753	Not Issued	161	02/14/2003	Skin Firming Anti-Aging Cosmetic Mask Compositions	GUPTA, SHYAM K.
10248817	Not Issued	161	02/21/2003	Boosting Tyrosinase Inhibiting Activity of Skin Whitening and Sunscreen Compositions	GUPTA, SHYAM K.
10248925	Not Issued	161	03/03/2003	Cold wax hair removal (depilatory) compositions	GUPTA, SHYAM K.
10249012	Not Issued	41	03/10/2003	Transparent Cold-Wax and Hot-Wax Depilatory Compositions with Three-Dimensional Suspended Particles	GUPTA, SHYAM K.
10249701	Not Issued	161	05/01/2003	Cosmetic and Pharmaceutical Masks Based on Ion-Pair Delivery System	GUPTA, SHYAM K.
10250045	Not Issued	161	05/30/2003	Hair Care and Nail Care Compositions Based on Ion-Pair Delivery System for Gender and Ethnic Selective Applications	GUPTA, SHYAM K.
10265000	Not Issued	161	10/04/2002	Ascorbic acid salts of organic bases with enhanced bioavailability for synergictic anti-aging and skin protective cosmetic compositions	GUPTA, SHYAM K.
10280519	Not Issued	161	10/25/2002	Niacinamide, niacin, and niacin esters based delivery systems for treating topical disorders of skin and skin aging	GUPTA, SHYAM K.
10290933	Not Issued	161	11/07/2002	Hydroxy acids based delivery systems for skin resurfacing and anti-aging compositions	GUPTA, SHYAM K.
10306948	Not Issued	83	11/29/2002	Trace Metals synergized copper nucleotides and copper glycosides for anti-aging and antiviral compositions	GUPTA, SHYAM K.
10307240	Not Issued	168	11/29/2002	Topical formulation including stabilized water-soluble and oil-soluble compositions	GUPTA, SHYAM K.
10394851	Not Issued	161	03/22/2003	Hydroxycitric acid derivatives for body slimming and tone firming	GUPTA, SHYAM K.

				compositions	
10418495	Not Issued	161	04/18/2003	Controlled-release nano-diffusion delivery systems for cosmetic and pharmaceutical compositions	GUPTA, SHYAM K.
10439349	Not Issued	161	05/15/2003	Ion-pair delivery system for cosmetic and pharmaceutical compositions	GUPTA, SHYAM K.
10604781	7179477	150	08/15/2003	COSMETIC DERMABRASION TREATMENT SYSTEM	GUPTA, SHYAM K.
10604999	Not Issued	71	08/29/2003	Antiaging Cosmetic Delivery Systems	GUPTA, SHYAM K.
10681938	Not Issued	41	10/09/2003	Liposomal delivery system for topical pharmaceutical, cosmeceutical, and cosmetic ingredients	GUPTA, SHYAM K.
10710011	Not Issued	30	06/11/2004	Zeolite based UV Absorbing and Sunscreen Compositions	GUPTA, SHYAM K.
10711136	Not Issued	30	08/26/2004	Zinc Zeolite Based Deodorants and Deodorizers	GUPTA, SHYAM K.
10711775	Not Issued	71	10/04/2004	Matrix metalloprotease (MMP) inhibitors and their application in cosmetic and pharmaceutical composition	GUPTA, SHYAM K.
10862037	Not Issued	30	06/05/2004	Skin whitening compositions based on hydroxyaryl alkyl ketones and their isosteric derivatives	GUPTA, SHYAM K.
10904665	Not Issued	30	11/22/2004	Topical Delivery System for Cosmetic and Pharmaceutical Agents	GUPTA, SHYAM K.
11126013	Not Issued	161	05/10/2005	Topical formulation including stabilized water-soluble and oil-soluble compositions	GUPTA, SHYAM K.
11163779	Not Issued	30	10/31/2005	Cosmetic Applications of Sucrose Polyhydroxy LactoneConjugates for Demabrasion,Depilation (Hair Removal), and wrinkles Reduction	GUPTA, SHYAM K.
11164709	Not Issued	25	12/02/2005	Cosmetic and Pharmaceutical Masks for Skin Improvement	GUPTA, SHYAM K.
11208306	Not Issued	30	08/18/2005	Cosmetic or pharmaceutical composition for skin care	GUPTA, SHYAM K.
11307729	Not Issued	30	02/18/2006	Concurrent Enhancement of Skin Penetration of Organic Base Active Agents and Organic Hydroxy Acid Active Agents as Their Ion-Pair Complexes	GUPTA, SHYAM K.

11307824	Not Issued	30	02/24/2006	Controlled-Release of Cosmetic and Pharmaceutical Agents via Osmotic Nano-Diffusion from Zeolite Cage Complexes	GUPTA, SHYAM K.
11308290	Not Issued	30	03/15/2006	Topical Delivery of Trace Metals for Enzyme Modulation	GUPTA, SHYAM K.
11309441	Not Issued	30	08/06/2006	Novel Hydroxy Acid Complexes for Antiaging and Skin Renovation	GUPTA, SHYAM K.
11615561	Not Issued	19	01/01/2007	Skin Condition Improvement Including Acne, Rosacea, and Topical Wounds by Artemisia Annua Extract via Iron Siderophore Trojan Horse Delivery System	GUPTA, SHYAM K.
11676284	Not Issued	25	02/17/2007	Sunscreen Safety and Efficacy Enhancement with Manganese Complexes via Urocanate Pathway	GUPTA, SHYAM K.
11684702	Not Issued	20	03/12/2007	Skin Whitening Methods and Compositions Based on Zeolite - Active Oxygen Donor Complexes	GUPTA, SHYAM K.
11716833	Not Issued	19	03/12/2007	Topical formulation including stabilized water-soluble and oil-soluble compositions	GUPTA, SHYAM K.
11760466	Not Issued	20	06/08/2007	Zinc Zeolite for the Treatment for Diaper Rash (Diaper Dermatitis)	GUPTA, SHYAM K.
06749324	Not Issued	166	06/27/1985	SUPERFATTED SOAPS	GUPTA, SHYAM K.

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Inventor Name Search Result

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Last Name = GUPTA
First Name = SHYAM

Application#	Patent#	Status	Date Filed	Title	Inventor Name
06894445	4704223	150	08/01/1986	SUPERFATTED SOAPS	GUPTA, SHYAM K.
06912978	Not Issued	168	09/29/1986	DETERGENT BAR COMPOSITIONS	GUPTA, SHYAM K.
07079948	Not Issued	161	07/31/1987	DETERGENT BAR COMPOSITIONS	GUPTA, SHYAM K.
07851463	5468887	150	03/13/1992	PRODUCTION OF FATTY ACID METHYL ESTERS AND SOAPS THEREFROM	GUPTA, SHYAM K.
60569568	Not Issued	159	05/10/2004	Topical skin treatment composition and method for applying the same, a system for creating the composition and conducting the method	GUPTA, SHYAM KIRTI

Inventor Search Completed: No Records to Display.

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